

TR 66917

JANUARY

1966

UNLIMITED

ROYAL AIRCRAFT ESTABLISHMENT

TECHNICAL REPORT No. 66017

AD 634909

THE PROTECTION OF
TANTALUM FROM OXIDATION
AT HIGH TEMPERATURES
WITH ALUMINIUM -
% TIN COATINGS

R. Moreton

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THE PROTECTION OF TANTALUM FROM OXIDATION AT HIGH TEMPERATURES
WITH ALUMINIUM - 50% TIN COATINGS

by

R. Moreton

SUMMARY

An Al - Sn coating was investigated as a protection against oxidation of tantalum sheet - a possible material for the combustion chambers of ramjets of hypersonic aircraft. Coatings containing equal parts by weight of Al and Sn were deposited from acetone slurries of the two metals and made impermeable by heating in vacuum for $\frac{1}{2}$ hr at 1050°C . The coatings, which then consisted of TaAl_3 covered with Al - Sn alloy, were assessed by oxidation tests, measurements of substrate hardening and mechanical tests. Tantalum was protected for up to $37\frac{1}{2}$ hr at 1500°C and $3\frac{1}{3}$ hr at 1600°C in air. Some self-healing occurred due to oxidation of molten Al - Sn alloy at defects in the outer oxide layer. Substrate hardening due to nitrogen penetration occurred in test-pieces where no signs of failure had been seen after heating in air at 1500°C . Tensile tests however showed that coated tantalum could be heated in air at 1500°C for up to 8 hr without loss of ductility at room temperature.

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1 INTRODUCTION

Tantalum has a melting point of 3000°C but in common with the other refractory metals it has poor resistance to oxidation at elevated temperatures. This is because the principal oxide Ta_2O_5 is porous and unprotective. Spoiser and St. Pierre¹ have reviewed the data available on the oxidation of tantalum and they quote the temperature at which the rate of oxidation becomes linear as being $700 - 750^{\circ}\text{C}$. The rate of recession of a tantalum surface heated in air at atmospheric pressure is reported to be 10^{-2}cm/hr at 1000°C . Tantalum does however retain a useful strength in vacuum up to comparatively high temperatures. At 1650°C for example the strength has been found² to be about 3500 lb/in^2 . Furthermore this can be improved by alloying, the strength of Ta-10% W alloy being about 12000 lb/in^2 at this temperature². Tantalum also has the advantages of ductility at room temperature and the fact that it can be welded more easily than tungsten or molybdenum. Therefore, although it has the rather high density of 16.6 gm/cc , it is potentially useful for high temperature applications such as ramjet flame tubes, if it can be protected from oxidation. Attempts to produce oxidation resistant tantalum alloys for use at high temperatures have had no success³ so attention has turned to developing protective coatings; this field has been reviewed recently by Gibeaut and Bartlett⁴ and Levinstein and Wlodek³.

One of the problems with protective coatings is to avoid failures due to thermal expansion mismatch between coating and substrate. An aluminium-tin coating has been developed⁵ in America which is reported to be self-healing at elevated temperatures. This coating, which can be applied by hot dipping, or by slurry application and heat treatment in vacuum, consists of a layer of tantalum aluminide, covered with aluminium - tin alloy which in turn has an outer skin of oxide. As the aluminium - tin alloy is liquid at operating temperatures, any small defects in this oxide are claimed to be repaired by oxidation of aluminium from the aluminium - tin layer. The oxidation test results quoted appeared to be very promising, for example aluminium - 50 wt % tin coatings on tantalum - 10% tungsten alloy were protective for 40 - 120 hr continuous testing at 1540°C in air.⁵ When these coatings were subjected to oxidation tests done in 1 hr cycles protection was obtained for at least 10 hr at temperatures up to 1621°C . Consequently it was decided that this type of coating was worth evaluating as a part of a programme of work on protective coatings.

Aluminium - 50 wt % tin coatings were applied to sheet tantalum by slurry application and heat treatment. These coatings were then subjected to oxidation tests and were examined by metallography and X-ray diffraction. Changes in hardness and ductility of tantalum substrates were measured after various heating times at 1500°C in air as it is known⁶ that solid solution of oxygen or nitrogen causes embrittlement. Similar tests were done in pure nitrogen to investigate the permeability of the coatings to nitrogen in the absence of oxygen.

2 EXPERIMENTAL

2.1 Preparation of test-pieces

Test-pieces were cut from 0.064 inch tantalum sheet obtained from Murox Limited. Two sizes were used (a) 10×1 cm for oxidation and tensile tests and (b) 1×1 cm for experiments involving microhardness measurements. The edges were rounded, the test-pieces were shot-blasted and then pickled in dilute hydrochloric acid.

2.2 Coating procedure

A slurry was made containing equal weights of aluminium and tin powders in acetone; in addition 2% of a nitrocellulose binder was also added. Coatings were then applied to the test-pieces either by painting or by dipping. After drying in air, the coatings were then heat-treated to 1050°C for $\frac{1}{2}$ hr in a vacuum ($1 - 3 \times 10^{-3}$ mmHg pressure). The aluminium and tin melted and thus formed uniform non-porous metallic coatings up to about 0.003 inch in thickness. Thicker coatings were obtained by repeating the process. It was found to be necessary to keep the furnace pressure below 3×10^{-3} mmHg. Higher pressures resulted only in loose powdery deposits due to surface contamination of the aluminium and tin powders.

2.3 Oxidation tests

The first series of oxidation tests were done at temperatures between $1200 - 1600^{\circ}\text{C}$ using resistance heating, the ends of the test-pieces being clamped in water-cooled copper blocks. The lower of the two blocks was free to move, being counterpoised, so as to allow expansion and contraction to occur. Testing was usually done in 2 hr cycles; the heating and cooling operations were controlled so as it takes a total time of 5 minutes, i.e. $2\frac{1}{2}$ minutes heating and $2\frac{1}{2}$ minutes cooling. Temperatures were measured with an optical pyrometer, no corrections for emissivity being made as the necessary information was not available. Coatings were considered to have failed when growths of Ta_2O_5 became visible, (see Fig.1).

The second series of test-pieces were oxidized in a horizontal platinum-wound tube furnace at 1500°C . This method was adopted as the small specimens used for the microhardness measurements could not readily be resistance heated and temperature gradients were to be avoided in the tensile test specimens. Temperatures were measured by means of a Pt/Pt - 13% Rh thermocouple. These tests, which were also done in 2 hr cycles, were continued for pre-determined periods of time and were then followed by tensile tests or hardness measurements, to see if embrittlement of the substrate had occurred. Several test-pieces were also heated at 1500°C in pure nitrogen to investigate nitrogen penetration of the coatings in the absence of oxygen. This was done using hardness measurements.

2.4 Examinations of coatings and substrates

Test-pieces, both before and after oxidation testing, were investigated by metallography, X-ray diffraction and analysis for oxygen and nitrogen. The composition of the coatings after deposition and heat treatment was also determined by chemical analysis.

Microhardness measurements were also made on the polished sections previously examined by metallography. A 41 gm load was used for all measurements, producing an indentation diagonal of about 25 microns for the tantalum sheet as received. The majority of the measurements were made 25 - 50 microns below the coating/metal interfaces. Mechanical tests were done using 2 inch gauge lengths, the tensile strengths and the elongations being measured.

3 RESULTS

Bright metallic coatings were obtained with thicknesses ranging between 0.004 - 0.011 inch as measured by micrometer. However as the coating heat treatments resulted in some formation of tantalum aluminide within the coating, the true thicknesses were slightly greater than those obtained by micrometer. Metallographic examinations showed that an addition of 0.001 inch was necessary. Thus the thicknesses quoted have been corrected by this amount. Fig.2(a) and (b) show a section through a coating after deposition, the coating consisting of Ta Al_3 covered with Al - Sn alloy.

The results of the first series of oxidation tests are given in Table 1. It can be seen that protection was obtained for 22 hr at 1200°C , $3\frac{1}{2}$ - $37\frac{1}{2}$ hr at 1500°C and $\frac{1}{2}$ - $3\frac{1}{3}$ hr at 1600°C . A typical coating failure at 1500°C is shown in Fig.1. Failures at 1500°C were localized and tended to start at sheet edges. On the other hand failures at 1600°C were more widespread, and after the coatings

had failed, tantalum oxidation occurred more rapidly. These results are less promising than those of Lawthers and Sama⁵ who claimed that Al - 50% Sn coatings were protective for at least ten 1 hr cycles at temperatures up to 1621°C in furnace tests. They also reported that the coatings survived 100 cycles to temperatures in the range 1200 - 1540°C using resistance heating. In these tests rapid cooling was achieved by means of an air blast. The substrate material in these tests was Ta - 10% W alloy. However Lawthers and Sama⁵ maintained that there was little difference between results with tantalum and Ta - 10% W alloy test-pieces.

Table 2 shows the tensile test results and Table 3 gives details of the microhardness measurements. It was found that substrate hardening could occur after heating coated test-pieces in air at 1500°C even though the coatings had appeared to be satisfactory. For example after one test lasting 6 hr the average hardness below the coating was approximately doubled. (See Table 3, Nos. 2 and 5). Similar tests done in nitrogen also resulted in hardened substrates, (see Table 3, Nos. 8 and 9). Some of the substrates were analysed to determine the oxygen and nitrogen contents, the results being given in Table 4. Substrate hardening was caused by nitrogen penetration of the coatings at 1500°C, whether the atmosphere was air or nitrogen. This is discussed in more detail in the next section.

4 DISCUSSION

X-ray diffraction indicated that a coating after heat-treatment contained tin and tantalum aluminide, $TaAl_3$. No aluminium was detected. However, chemical analysis of a similar coating using hydrochloric acid to remove any aluminium and tin did show aluminium to be present, the Al - Sn ratio being 0.22 by weight. This ratio was less than one because the hydrochloric acid had not removed the aluminide layer. A second analysis involving the removal of the coating with hot aqua regia and concentrated sulphuric acid resulted in a ratio of 1.04. This is in good agreement with the slurry ratio of 1.00 for Al/Sn.

The changes that took place in the coatings during the oxidation tests were investigated by X-ray diffraction. Photomicrographs of coatings after deposition and after 8 hr in air at 1500°C are shown in Figs. 2, 3 and 4. It was found that after 6 hr in air at 1500°C a coating contained an outer layer of oxide, some tin and the σ (Ta - Al) phase. The oxide contained $\alpha-Al_2O_3$ and another unidentified material. By analogy with the σ phase in the Nb - Al system⁷ the σ phase found in the coating probably consisted of Ta_2Al . Thus it appeared that all the $TaAl_3$ had been converted to Ta_2Al during the test. This

is in agreement with Lawthers and Sama⁵ who found the phase Ta_2Al in an Al - 50% Sn coating on Ta - 10% W alloy after 10 hr at $1482^{\circ}C$.

During the heating-up periods to the testing temperatures small beads of molten Al - Sn alloy, usually up to about 1 mm diameter were seen to exude from the coatings. These were quickly oxidized with small pieces of oxide sometimes falling from the test-pieces. The eventual coating failures did not seem to be directly associated with the points where beads of molten alloy had been seen. However the formation of these beads did result in losses of Al - Sn alloy from the coatings and it was noted that less alloy exuded during the heating-up periods the longer the tests had been running. Thus coating failures became more likely as the amounts of Al - Sn alloy available for the repair of defects in the oxide were reduced. In addition to this the concentration of aluminium in the Al - Sn layers was reduced during testing by reaction with the substrate. Lawthers and Sama⁵ maintained that it was important that a protective oxide was formed on Al - Sn coatings before the concentration of free aluminium was reduced by reaction with the substrate. However, they suggested that after the $TaAl_3$ had been converted to Ta_2Al , the concentration of aluminium in the Al - Sn alloy layers was still sufficient to provide self-healing of defects in the oxide layers.

The foregoing discussion suggests that better protection should be obtained with thicker coatings. In this work it was found that coatings up to 0.012 inch could be deposited on 1 x 1 cm sheet test-pieces. In the case of 10 x 1 cm test-pieces, the dripping of molten Al - Sn alloy from the substrates during heat-treatment made it difficult to apply uniform coatings thicker than 0.009 inch. A recent approach to the problem of obtaining thick uniform coatings involved the addition of refractory metal powders to the Al - Sn slurries⁴. During heat-treatment the refractory metal powder reacted with some of the aluminium to form aluminide particles. This was intended to prevent the molten Al - Sn layer dripping from the substrate by increasing the viscosity. Several compositions were tried, an example being the addition of tantalum powder to a slurry of Al - 50% Sn powders. The amount of tantalum used was sufficient to produce 10% of $TaAl_3$ particles in the coatings. The protection this coating gave to Ta - 10% W alloy test-pieces was 6 - 7 hr at $1540^{\circ}C$, when tested in 1 hr cycles. The coating thicknesses obtained were only 0.006 inch however, i.e. they were not as thick as some of those applied here.

The suggestion that thicker coatings are more protective is supported experimentally by the microhardness measurements. For example the average substrate hardness below a 0.007 inch coating was approximately doubled after 6 hr at $1500^{\circ}C$ in air whereas the average hardness beneath a 0.012 inch coating

was virtually unchanged after 10 hr at 1500°C, (see Table 3 Nos. 5 and 7).

When the microhardness measurements were being made, it was noticed that substrate hardening tended to be more pronounced near the sheet edges. This may be due to the formation of fissures in the coatings, as shown in Fig. 4, which was taken from a section through a test-piece after 8 hr at 1500°C in air. It can be seen that at a point where the substrate hardness was 235 V.H.N. fissures were present although they did not appear to penetrate right through to the substrate. For comparison Fig. 3 shows another region of the same section where no hardening had occurred. These fissures were probably caused by thermal expansion mismatch, the expansion coefficients (α) of the various components involved, being as follows:-

$$\begin{aligned}\alpha(\text{Ta}) &= 7.6 \times 10^{-6}/^{\circ}\text{C} \quad (20 - 1500^{\circ}\text{C})^8 \\ \alpha(\text{Al}) &= 27.3 \times 10^{-6}/^{\circ}\text{C} \quad (20 - 500^{\circ}\text{C})^3 \\ \alpha(\text{Sn}) &= 24.2 \times 10^{-6}/^{\circ}\text{C} \quad (20 - 200^{\circ}\text{C})^9 \\ \alpha(\text{Al}_2\text{O}_3) &= 9.7 \times 10^{-6}/^{\circ}\text{C} \quad (20 - 1500^{\circ}\text{C})^8\end{aligned}$$

The expansion coefficients of TaAl_3 and Ta_2Al , although not known, are probably greater than that of tantalum.

Thus, on cooling, fissures or cracks are likely to occur due to tensile stresses resulting from the larger contractions of the coatings.

Another feature of the hardness measurements was that heating in nitrogen at 1500°C caused more substrate hardening than similar exposures in air. For example, 6 hr in nitrogen resulted in an average hardness below the coating of 478 V.H.N. as compared with 286 V.H.N. after 6 hr in air, (see Table 3 Nos. 5 and 9). It was found, by determining the oxygen and nitrogen contents of some hardened substrates, that the hardening was due to nitrogen penetration whether the test-pieces had been heated in nitrogen or air, (see Table 4). For example, 6 hr in nitrogen at 1500°C resulted in a nitrogen increase from about 0.003 at.% to 0.401 at.%. Another test-piece heated for 6½ hr in air at 1500°C was found to have a nitrogen content of 0.142 at.% but with no significant increase in oxygen content. The hardness variation across this test-piece is shown in Fig. 5. The main reason why hardening was more pronounced in nitrogen was that an appreciable aluminium oxide skin had not been formed.

Variations of hardness with distance below the coatings are shown in Fig. 6 and 7 after heating to 1500°C in air and nitrogen respectively. The fact that hardening could occur 600 microns, or 0.024 inch below the coatings indicated that the tensile strength and ductility of the material could also be affected.

Tensile tests were therefore done after heating test-pieces in air at 1500°C . It was found that the coatings were capable of preventing loss of ductility for up to 8 hr although reduction in strength occurred. This strength reduction was considered to be partly due to the conversion of tantalum to tantalum aluminides. It was found that after 8 hr at 1500°C the substrate thickness had been reduced to about 0.056 inch. The thickness after the coating had been applied was about 0.062 inch. Another cause of the loss in strength was that appreciable grain growth had occurred.

Although none of the oxidation tests described here were done at reduced pressures, it should be mentioned that aluminium-tin coatings have been found to fail more rapidly when tested under certain high temperature/low pressure conditions⁴. For example, about 70 wt % of a coating was found to have been lost after $\frac{1}{2}$ hr at 1427°C in air at 1.5 mmHg pressure. This was considered to be due to evaporation of the liquid Al - Sn phase. On the other hand however the coatings were found to be stable when tested at 3 mmHg air pressure at 1427°C and also at 6 mmHg pressure at 1538°C .

5 CONCLUSIONS

(1) Metallic, non-porous coatings up to 0.012 inch were obtained on tantalum sheet by applying slurries containing equal weights of aluminium and tin powders in acetone, followed by drying in air and vacuum heat-treatment for $\frac{1}{2}$ hr at 1050°C .

(2) X-ray diffraction and chemical analysis showed that the freshly formed coatings consisted of tantalum aluminide TaAl_3 covered with an aluminium-tin alloy.

(3) The coatings were found to protect tantalum sheet test-pieces, 0.064 inch in thickness, for up to $37\frac{1}{2}$ hr at 1500°C and $3\frac{1}{3}$ hr at 1600°C in air. The average lifetimes at 1500°C and 1600°C were $13\frac{1}{2}$ hr and $1\frac{1}{3}$ hr respectively.

(4) During the oxidation tests the tantalum aluminide TaAl_3 was converted to the lower aluminide Ta_2Al by reaction with the substrate.

(5) The oxidation resistance of the coatings was a consequence of the formation of an outer oxide layer which contained alumina. The coatings were self-healing due to oxidation of molten aluminium-tin alloy at defects in this oxide layer.

(6) Hardening of the substrate sometimes occurred in test-pieces when no signs of failure had been seen after heating in air at 1500°C . It was found that the thicker coatings were more effective in preventing hardening.

(7) Estimation of the nitrogen content of a hardened substrate after an oxidation test lasting $6\frac{1}{2}$ hr at 1500°C showed that the hardening in air was due to nitrogen permeation.

(8) It was confirmed that the coatings were slightly permeable to nitrogen at 1500°C in that heating for 6 hr in a pure nitrogen atmosphere caused substrate hardening due to an increase in nitrogen content.

(9) Tensile tests done on coated test-pieces showed that the coatings were capable of preventing loss of ductility at room temperature for up to 8 hr heating in air at 1500°C .

ACKNOWLEDGEMENTS

The author is grateful to Mr. D. Clark who examined the coatings by X-ray diffraction and to Mr. M. S. Binning who did the tensile tests.

Table 1. Oxidation test results for tantalum sheet protected with aluminium - 50% tin coatings

Coating thickness (in)	Testing temperature (°C optical temperature)	Lifetime (hr)	No. of cycles	Remarks
0.005	1200	22	8	Coating became loose at ends where the temperature was about 800°C. No substrate hardening had occurred however. (see Table 3, No.10).
0.006	1500	6½	3	Failed near the top grip. Although the coating in the centre appeared to have been protective substrate hardening up to 292 V.H.N. was found to have occurred there. (see Table 3, No.11 and Fig.5)
0.009	1500	3½	2	Tested in a tube furnace. Failed on an edge as shown in Fig.1. Temperature measured by thermocouple
0.008	1500	6¼	4	Failed near the centre on an edge
0.009	1500	37½	19	Failed in the centre
0.005	1600	½	1	Widespread failure in the centre
0.005	1600	3¼	2	Widespread failure in the centre
0.009	1600	1	1	Widespread failure in the centre
0.009	1600	½	1	Widespread failure in the centre

* Test-pieces 10 x 1 cm were resistance heated in still air

Table 2 - Results of mechanical tests on coated test-pieces

Coating thickness (in)	Subsequent treatment of test-pieces*	No. of cycles	Tensile strength at room temperature (lb/in ²)	% Elongation (2 inch gauge length)
-	(Shot blasted but not coated)	-	50620	29
0.005	None	-	45470	26
0.008	Heated for 4 hr at 1500°C in air without visible failure	2	28000	29
0.009	Heated for 8 hr at 1500°C in air without visible failure	4	25540	25

*Test-pieces were 10 x 1 cm

Table 3 - Microhardness measurements on sections through tantalum test-pieces

No.	Type of test-piece (cm)	Coating thickness (in)	Subsequent treatment of test-pieces	No. of cycles	Microhardness, V.H.N. (Kg/mm ²)		No. of results
					Range	Average	
1	1 x 1	-	(Tantalum sheet as received)	-	141-157	149	10
2	1 x 1	0.006	None	-	132-140	136	10
3	1 x 1	0.007	Heated for 2 hr at 1500°C in air*	1	132-158	144	10
4	1 x 1	0.006	Heated for 4 hr at 1500°C in air*	2	147-244	182	10
5	1 x 1	0.007	Heated for 6 hr at 1500°C in air*	3	222-360 (see also Fig.6)	286	10
6	1 x 1	0.009	Heated for 8 hr at 1500°C in air*	4	129-235	162	12
7	1 x 1	0.012	Heated for 10 hr at 1500°C in air*	5	129-193	146	12
8	1 x 1	0.008	Heated for 4 hr at 1500°C in nitrogen*	2	386-456 (see also Fig.7)	409	10
9	1 x 1	0.003	Heated for 6 hr at 1500°C in nitrogen*	3	396-645	478	12
10	10 x 1	0.005	Heated for 22 hr at 1200°C optical temperature in air	8	Measurements made at an end where coating appeared to be loose.	134-146	6
					Measurements made in the middle of the test-piece*	131-149	10

*No sign of coating failure was seen

Measurements were made 25-50 microns below the coating/metal interfaces

Table 4 - Nitrogen and oxygen contents of some hardened substrates

Coating thickness (in)	Subsequent treatment of test-pieces	Microhardness of substrate, V.H.N. (Kg/mm ²)	Type of test-piece (cm)	Nitrogen content (atomic %)	Ratio: nitrogen content/initial nitrogen content	Oxygen content (atomic %)	Ratio: oxygen content/initial oxygen content
0.010	None	-	1 x 1	0.003	-	0.029	-
0.006	6½ hr at 1500°C optical temperature in air	120-292 (see Table 3 No.11 and Fig.5)	10 x 1*	0.142	47	0.039	1.3
0.008	6 hr at 1500°C in nitrogen	396-645 (see Table 3 No.9)	1 x 1	0.401	134	0.052	1.8

/ after removal of the coatings

* the piece analysed was 1 x 1 cm taken from the centre

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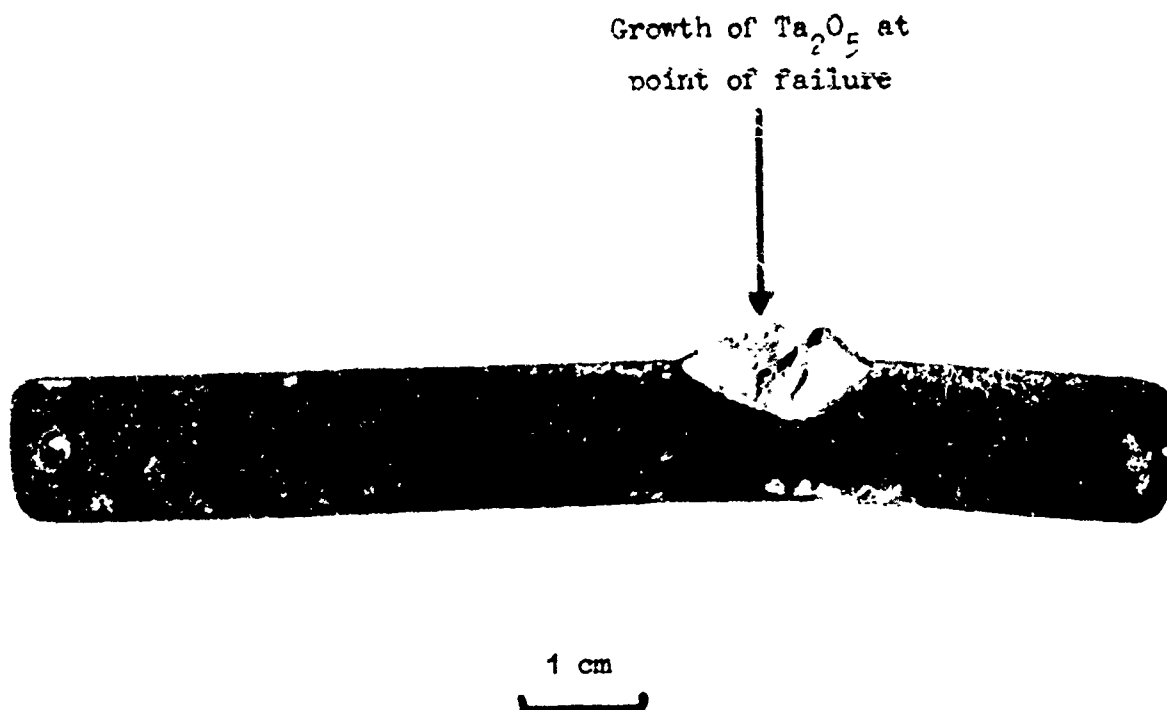


FIG 1 FAILURE OF AN ALUMINIUM-50% TIN COATING ON TANTALUM AFTER
3½ HR AT 1500°C IN AIR (X 1.5)

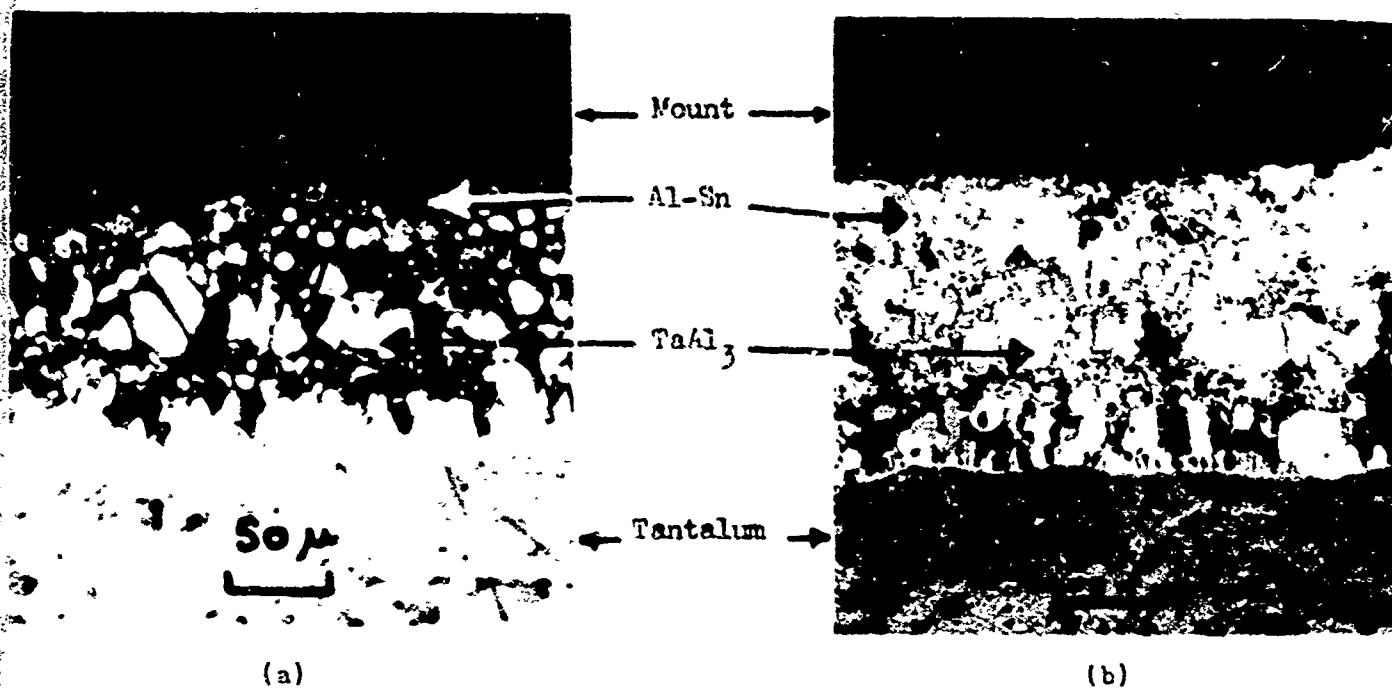


FIG.2(a) SECTION THROUGH A TANTALUM TEST-PIECE AFTER APPLYING
TWO COATINGS OF ALUMINIUM-50% TIN (X 250)
(b) SAME FIELD OF VIEW SEEN THROUGH PARTIALLY CROSSED
NICOLS (X 250)

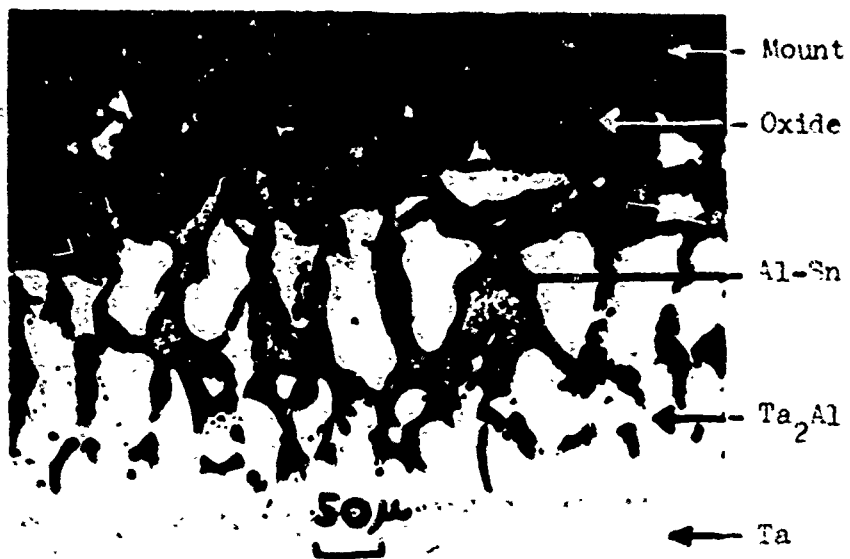


FIG.3 FIELD OF VIEW WHERE NO SUBSTRATE HARDENING HAD OCCURRED:
MICROHARDNESS WAS 132 V H N (X 150)

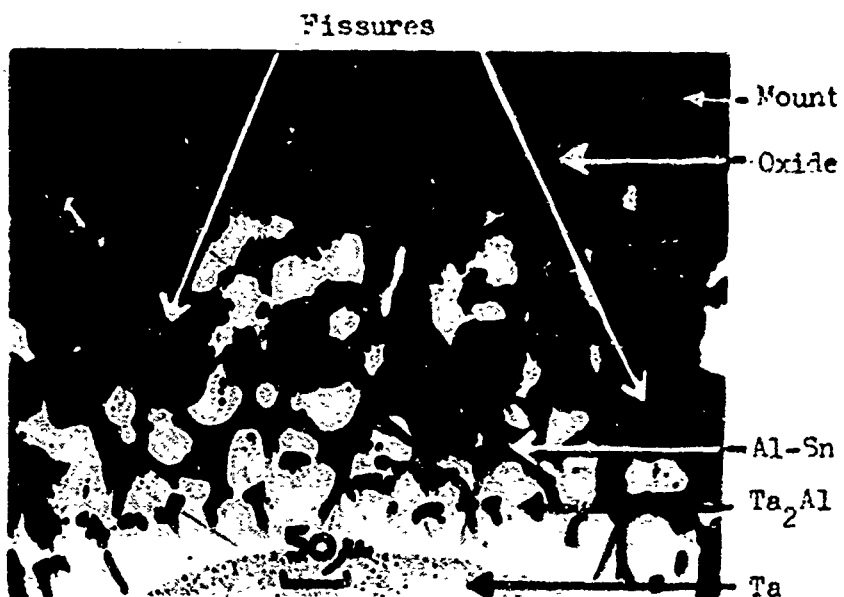


FIG.4 FIELD OF VIEW SHOWING AN EDGE WHERE LOCALIZED HARDENING
OF THE SUBSTRATE HAD OCCURRED: MICROHARDNESS
WAS 235 V.H.N. (X 150)

FIG.3 & 4 SECTION THROUGH A TANTALUM TEST-PIECE COATED WITH
ALUMINIUM-50% TIN AND HEATED IN AIR FOR 8 HR
AT 1500°C WITHOUT FAILURE

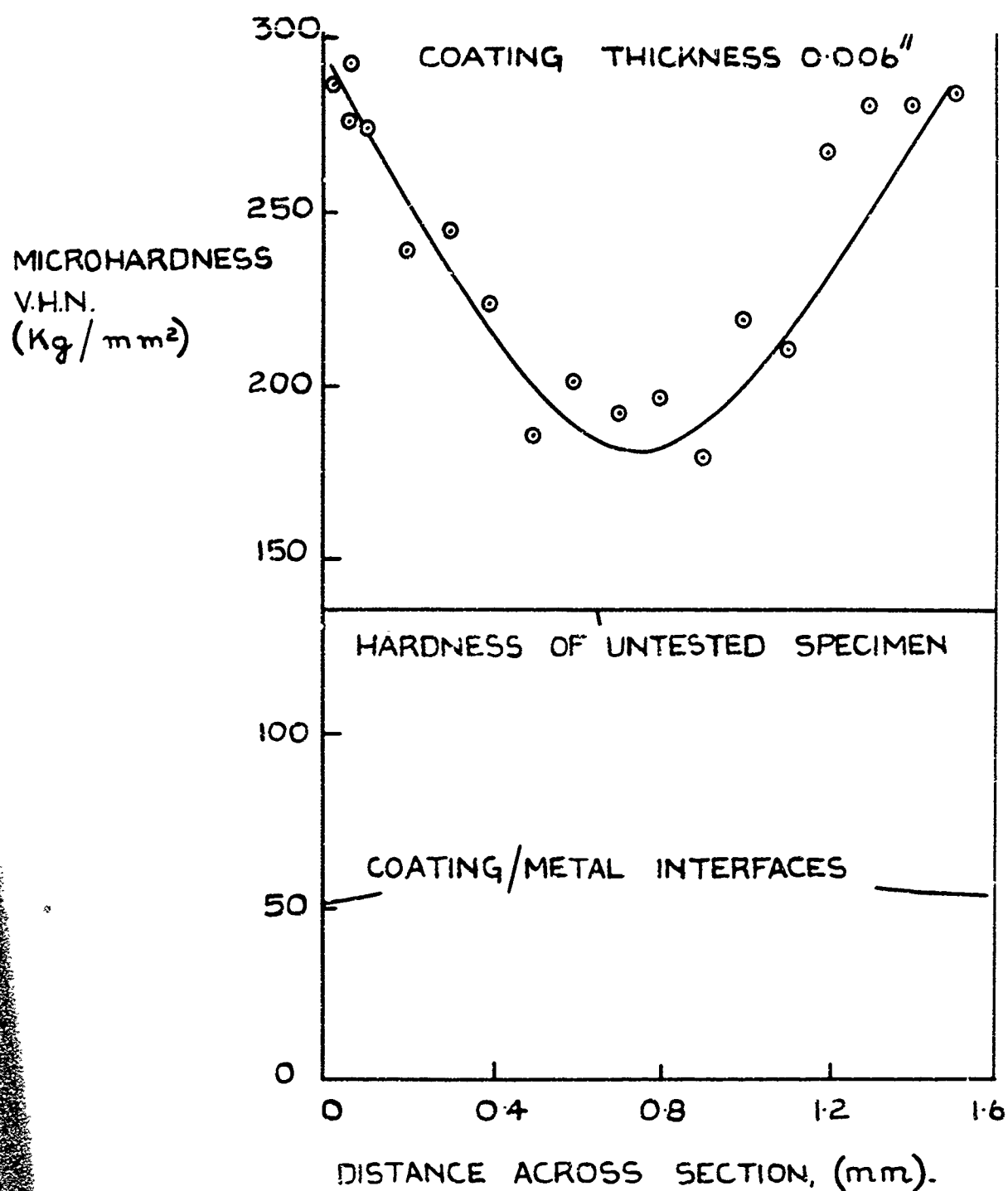


FIG. 5. VARIATION OF MICROHARDNESS ACROSS THE THICKNESS OF A TEST-PIECE COATED WITH ALUMINIUM-50% TIN AFTER AN OXIDATION TEST LASTING 6½ hr AT 1500°C OPTICAL TEMPERATURE IN AIR

CPM 014-900022

Fig. 6 & 7

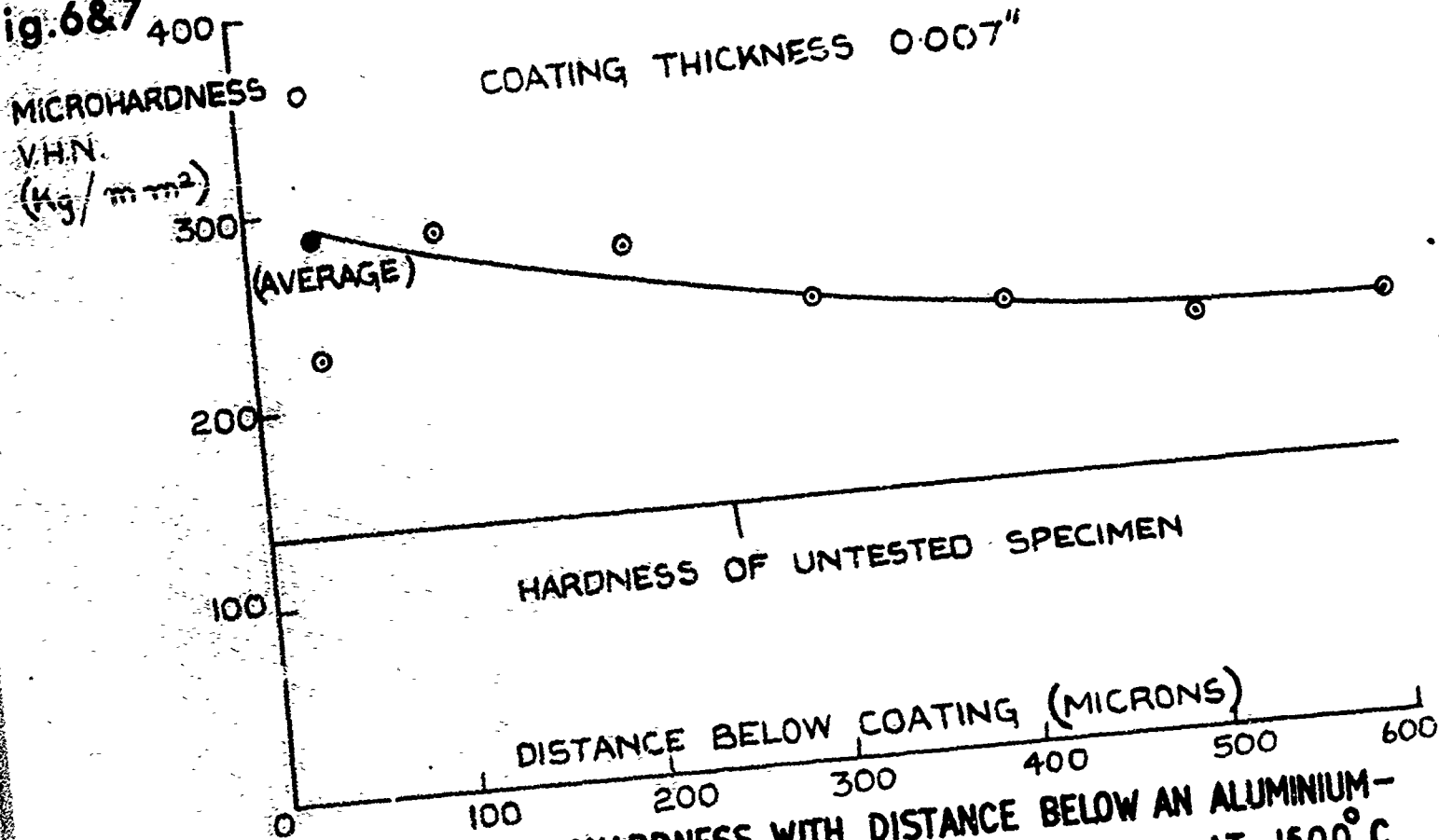


FIG. 6. VARIATION OF MICROHARDNESS WITH DISTANCE BELOW AN ALUMINIUM-50% TIN COATING ON TANTALUM AFTER HEATING FOR 6 hr AT 1500°C IN AIR WITHOUT FAILURE

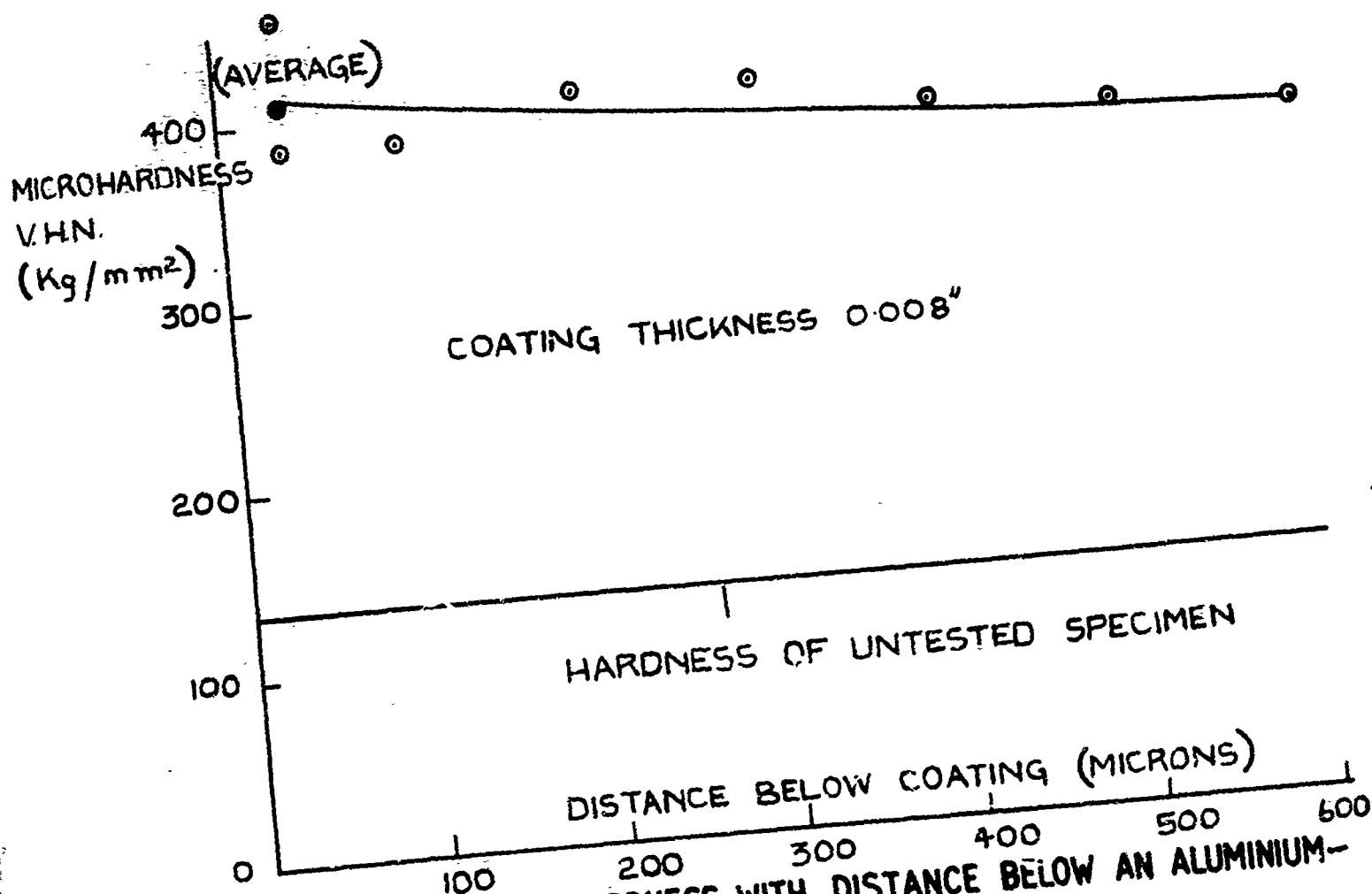


FIG. 7. VARIATION OF MICROHARDNESS WITH DISTANCE BELOW AN ALUMINIUM-50% TIN COATING ON TANTALUM AFTER HEATING FOR 4 hr AT 1500°C IN NITROGEN